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IS 6031 (1997): Calcium Propionate, Food Grade [FAD 8: Food Additives]

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कैल्सियम प्रोपायोनेट, खाद्य ग्रेड — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

CALCIUM PROPIONATE, FOOD GRADE — SPECIFICATION

(*First Revision*)

ICS 67.220.20; 71.080.99

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1997 **Price Group 3**

Food Additives Sectional Committee, FAD 8

FOREWORD

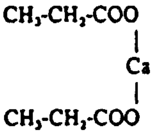
This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties, etc, of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards was, therefore, prepared to cover purity and identification of these substances. These standards would help in checking purity, which requires to be checked at the stage of manufacture, for it is extremely difficult to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies.

Calcium propionate is an anti-roping agent and a mould inhibitor. It is also permitted for bread under the *Prevention of Food Adulteration Rules*, 1955. These rules, *inter-alia* prescribe:

> 'The listed food additives permitted for use in certain foods shall be sold only under the BIS Certification Mark.' Calcium propionate, food grade is one among the listed additives.

Chemical Names and Formula — Calcium propionate or calcium propanoate. Its empirical formula is $C_6H_{10}O_4Ca$. Its molecular weight is 186.23. Structural formula is:



$$\begin{array}{r} CH_3\text{-}CH_2\text{-}COO \\ | \\ Ca \\ | \\ CH_3\text{-}CH_2\text{-}COO \end{array}$$

STRUCTURAL FORMULA

This standard was first published in 1971 and is being revised to incorporate the following additions/ changes:

a) to provide description clause, including solubility, separately from requirements to keep it in line with Food Chemical Codex, NRC.

b) to upgrade the standard by providing requirements for fluoride and magnesium.

c) to substitute the requirement of lead by heavy metals and corresponding change in test method.

d) to provide for 'directions for storage' and 'Best before date'.

Considerable assistance has been derived from the following publications in preparing this standard. In the preparation of this standard, considerable assistance has been derived from:

> Compendium of Food Additive Specifications, Volume 1, Joint FAO/WHO Expert Committee on Food Additives (JECFA), 1992.
>
> Food Chemical Codex (FCC), Third Edition. National Academy of Science, National Research Council, Washington, DC.

This standard is deviating from JECFA (FAO/WHO) with respect to *p*H requirement and from FCC with respect to matter insoluble in water.

FAO/WHO requirement for *p*H is 6-9 and FCC requirement for matter insoluble in water is 0.2 percent by mass, *Max*.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CALCIUM PROPIONATE, FOOD GRADE — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and methods of tests for calcium propionate, food grade.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1699 : 1995	Methods of sampling and test for synthetic food colours (*second revision*)
2362 : 1993	Method for determination of water by the Karl Fischer method (*second revision*)

3 DESCRIPTION

It shall be in the forms of white crystals or crystalline solid or possessing a faint odour of propionic acid. The material shall be freely soluble in water.

NOTE — The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity, and dependence must be placed on other specifications.

4 REQUIREMENTS

4.1 Identification Tests

4.1.1 Five percent solution of material gives positive test for calcium. The presence of the calcium may be demonstrated by reacting a solution of calcium propionate with ammonium oxalate solution. The white precipitate formed is soluble in hydrochloric acid, but insoluble in acetic acid.

4.1.2 Upon ignition at a relatively low temperature, it yields an alkaline residue which effervesces with acids.

4.1.3 When warmed with sulphuric acid, propionic acid is evolved, recognizable by its odour.

4.2 The *p*H of the 10 percent (*m/v*) solution of the material at 25 ± 2°C, shall be between 7 and 9.

4.3 The material shall also conform to the requirements given in Table 1.

Table 1 Requirements for Calcium Propionate

Sl No.	Characteristic	Limit	Method of Test, Ref to	
			Annex of This Standard	Other Standard
(1)	(2)	(3)	(4)	(5)
i)	Purity as $C_6H_{10}O_4Ca$, percent by mass, on dry basis, *Min*	98	A	—
ii)	Moisture, percent by mass, *Max*	5.0	B	—
iii)	Matter insoluble in water, percent by mass, *Max*	0.3	C	—
iv)	Arsenic (as As), mg/kg, *Max*	3	—	15 of IS 1699
v)	Heavy metals (as Pb), mg/kg, *Max*	10	—	16 of IS 1699
vi)	Iron (as Fe), mg/kg, *Max*	50	D	—
vii)	Fluoride, mg/kg, *Max*	10	E	—
viii)	Magnesium (as MgO)	To Pass the test (about 0.4%)	F	

5 PACKING, STORAGE AND MARKING

5.1 Packing

The material shall be securely packed in well-filled containers with minimum access to light and moisture. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

5.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

5.3 Marking

Each container shall be legibly and indelibly marked with the following information:

a) Name of the material including the words 'Food Grade';
b) Name and address of the manufacturer;
c) Net content when packed;
d) Batch or code number;
e) Instructions for storage;
f) Expiry/best before date; and
g) Any other requirements as specified under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977 and *Prevention of Food Adulteration Rules*, 1955.

5.3.1 *BIS Certification Marking*

The containers may also be marked with the BIS Standard Mark.

5.3.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 The representative samples of the material shall be drawn according to the method prescribed in 4 of IS 1699.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF CALCIUM PROPIONATE CONTENT

A-0 GENERAL

A-0.1 Three methods have been specified. Any of them could be used depending upon the facilities available. However, Method III shall be used as referee method.

A-1 METHOD I

A-1.1 Reagents

A-1.1.1 *Phosphoric Acid* — 50 percent.

A-1.1.2 *Sodium Hydroxide Solution* — 1 N.

A-1.1.3 *Phenolphthalein Indicator*

Dissolve 2 g of phenolphthalein in 60 ml of 90 percent ethanol and add sufficient water to make 100 ml.

A-1.2 Procedure

Weigh 3.00 g of sample, into a distillation flask and add 200 ml of phosphoric acid. Heat to boiling for 2 h and collect the distillate. During distillation keep the volume in the flask at about 200 ml by adding water using a dropping funnel. Titrate the distillate with 1 N sodium hydroxide solution using phenolphthalein as indicator. Each millilitre of 1 N sodium hydroxide solution corresponds to 0.093 11 g of calcium propionate.

A-2 METHOD II

A-2.1 Reagents

A-2.1.1 *Sodium Hydroxide*

Dissolve 4.3 g of sodium hydroxide in water to make 100 ml.

A-2.1.2 *Hydroxynaphthol Blue Indicator*

A-2.1.3 *Disodium Ethylenediaminetetraacetate* (*EDTA*) — 0.05 M, that is, 16.81 g of $C_{10}H_{14}N_2Na_2O_5$ per litre.

A-2.2 Procedure

A-2.2.1 Dissolve about 400 mg of the sample, accurately weighed, in 150 ml of water, add 15 ml of sodium hydroxide and 300 mg of hydroxynaphthol blue indicator. Titrate with disodium ethylenediaminetetraacetate until the solution is clear blue in colour. Each millilitre of 0.05 M disodium ethylenediaminetetraacetate is equivalent to 9.311 mg of $C_6H_{10}O_4Ca$.

A-3 METHOD III

A-3.1 Apparatus

A-3.1.1 *p*H-meter with glass calomel electrode assembly.

A-3.1.2 *Mechanical Stirrer*

A-3.1.3 *Wide-Rimmed Beaker* — 200 ml.

A-3.2 Reagents

A-3.2.1 *Glacial Acetic Acid*

A-3.2.2 *Perchloric Acid* — 0.1 N.

A-3.3 Procedure

A-3.3.1 Tare a clean, dry weighing bottle to an accuracy of ± 0.2 mg and weight to the nearest mg, 0.4 g of the sample, previously dried at 120 for 2 h, into it, slide the opened weighing bottle into a 200 ml wide-rimmed beaker containing 50 ml of glacial acetic acid and swirl to effect solution. Place a mechanical agitator and a glass calomel electrode assembly into the beaker in such a manner that the weighing bottle is lodged behind the electrodes and does not move with the agitation. Set the *p*H-metre on + mV circuit and titrate the sample with 0.1 N perchloric acid. Add the perchloric acid in large increments until the mV change with each addition shows that the titration is nearing the end point the deflection of the needle becomes noticeable. Then reduce the added fraction to – 0.1 ml and take mV-circuit readings until there is a further decrease in the mV changes after each addition. Plot the curve obtained from the millilitres added against the mV-readings and determine the quantity of the titrant corresponding of half-way up the steepest gradient.

$$\text{Percent calcium propionate} = \frac{V \times N \times 0.093\,11}{W} \times 100$$

where

V = ml of the perchloric acid,
N = exact normality of the perchloric acid, and
W = mass (in g) of the sample.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF MOISTURE

B-0 GENERAL

B-0.1 Oven drying and Karl Fischer methods have been specified. In case of dispute Karl Fischer method shall be used.

B-1 OVEN DRYING METHOD

B-1.1 Apparatus

B-1.1.1 *Drying Oven* — capable of operating at 110°C.

B-1.1.2 *Flat Bottom Dish* — made of nickel or other suitable material, 7 to 8 cm in diameter, not more than 2.5 cm deep.

B-1.2 Procedure

Weigh accurately about 2 g of the material in the tared dish. Place the dish containing the sample in the oven maintained at 110 ± 1°C. Remove the dish from the oven after 4 h. Cool in a desiccator and weigh. Dry again for 30 min, cool and weigh. Continue until two successive weighings do not differ by more than 1 mg.

B-1.3 Calculate the loss on drying percent by mass.

B-2 KARL FISCHER METHOD

B-2.1 Moisture may be determined by Karl Fischer method described in IS 2362 using 0.5 g of the material.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF MATTER INSOLUBLE IN WATER

C-1 APPARATUS

C-1.1 Crucible — sintered glass or porcelain of porosity G 4.

C-2 PROCEDURE

C-2.1 Weigh, to the nearest 0.1 g, 25.0 g of the material and dissolve in 250 ml of water contained in a 400-ml beaker. Stir until solution is complete and then allow to stand for 1 h. Filter the solution through the sintered crucible, previously dried and weighed to the nearest 0.2 mg, transferring any insoluble matter into the crucible with a jet of water. Wash the crucible with several portions of water. Dry the crucible in an oven at 100 to 105°C for 1 h, cool in a desiccator and weigh. Continue until two successive weighings do not differ by more than 0.2 mg.

C-3 CALCULATION

C-3.1 Matter insoluble in water, percent by mass $= \frac{M_1 \times 100}{M_2}$

where

M_1 = mass, in g, of insoluble residue; and

M_2 = mass, in g, of sample taken.

ANNEX D

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF IRON

D-1 APPARATUS

D-1.1 Nessler Cylinders — 50 ml, two matched.

D-1.2 Crucible — platinum or any suitable crucible.

D-2 REAGENTS

D-2.1 Nitric Acid — concentrated, sp gr = 1.42, free from iron.

D-2.2 Hydrochloric Acid — approximately 1 N solution.

D-2.3 Hydroxylammonium Chloride Solution — 10 g/l.

D-2.4 2,2′-bipyridyl Solution — 1 g/l. Dissolve 0.1 g of the reagent in 50 ml of water containing 2 ml of hydrochloric acid and dilute to 100 ml with water.

D-2.5 Ammonium Acetate — 200 g/l solution.

D-2.6 Standard Iron Solution

Dissolve 7.022 g of ammonium ferrous sulphate in a mixture of 600 ml of water and 350 ml of concentrated sulphuric acid, sp gr = 1.84. Dilute to 1 000 ml with water and further dilute 10 ml of the solution so obtained to 1 000 ml with water. One millilitre of the diluted solution contains 10 μg of iron. This final solution should be freshly prepared.

D-3 PROCEDURE

D-3.1 Weigh, to the nearest 0.01 g, 2 g of the sample, transfer to the platinum or any suitable crucible and carefully ignite. Moisten the residue with a few drops of the nitric acid and carefully heat to dryness. Dissolve the residue in about 10 ml of water containing 2 ml of the hydrochloric acid solution and transfer the solution to one of the Nessler cylinders, using about 15 ml of wash water.

D-3.1.1 Add 5 ml of hydroxylammonium chloride solution and 10 ml of the ammonium acetate solution, mix, add 3 ml of the 2,2'- bipyridyl solution and dilute to the mark with water. Mix thoroughly and allow to stand for 10 min.

D-3.1.2 At the same time carry out a comparison test (equivalent to 100 parts per million by mass of iron) using 10 ml of the standard iron solution in place of the test solution. Compare the colours of the two solutions and note whether the colour of the sample solution is greater than that of the comparison solution.

ANNEX E

[*Table* 1, *Sl No.* (vii)]

TEST FOR FLUORIDE

E-0 GENERAL

E-0.1 There are two methods and either of them could be used.

E-1 METHOD I THORIUM NITRATE COLORIMETRIC METHOD

Caution: When applying this test to organic compounds, the temperature at which the distillation is conducted must be rigidly controlled at all times to the recommended range of 135° to 140° to avoid the possibility of explosion.

NOTE — To minimize the distillation blank resulting from fluoride leached from the glassware, the distillation apparatus should be treated as follows: Treat the glassware with hot 10 percent sodium hydroxide solution, followed by flushing with tap water and rinsing with distilled water. At least once daily, treat in addition by boiling down 15 to 20 ml of dilute sulphuric acid (1 in 2) until the still is filled with fumes; cool, pour off the acid, treat again with 10 percent sodium hydroxide solution, and rinse thoroughly.

E-1.1 Reagents

E-1.1.1 *Perchloric Acid*

E-1.1.2 *Silver Nitrate Solution* — (1 in 2)

E-1.1.3 *Sodium Alizarin Sulfonate Solution* — (1 in 1 000).

E-1.1.4 *Hydroxylamine Hydrochloride Solution* — (1 in 4 000).

E-1.1.5 *Sodium Hydroxide* — 0.05 N or 1 N.

E-1.1.6 *Hydrochloric Acid* — 0.1 N.

E-1.1.7 *Thorium Nitrate Solution* — (1 in 4 000).

E-1.1.8 *Sodium Fluoride Solution* (10 μg F per ml).

Transfer 2.210 g of sodium fluoride, previously dried at 110° for 2 h and accurately weighed, into a 400-ml plastic beaker, add 200-ml of water and stir until dissolved. Quantitatively transfer this solution into a 1 000-ml volumetric flask with the aid of water, dilute to volume with water and mix. Store this stock solution in a plastic bottle. On the day of use, transfer 10 ml of the stock solution into a volumetric flask dilute to volume with water and mix.

Dilute sulphuric acid (1:2).

E-1.2 Procedure

Place 1.67 g sample and 30 ml of water in a 125-ml distillation flask having a side arm and trap. The flask is connected with a condenser and carries a thermometer and a capillary tube, both of which must extend into the liquid. Slowly add, with continuous stirring, 10 ml of perchloric acid, and then add 2 or 3 drops of silver nitrate solution (1 in 2) and a few glass beads. Connect a small dropping funnel or a steam generator to the capillary tube. Support the flask on an asbestos mat with a hole that exposes about one-third of the flask to the flame. Distil until the temperature reaches 135°. Add water from the funnel or introduce steam through the capillary, maintaining the temperature between 135° and 140° at all times. Continue the distillation until 100-ml of distillate has been collected. After the 100-ml portion (Distillate A) is collected, collect an additional 50-ml portion of distillate (Distillate B) to ensure that all of the fluorine has been volatilized.

Place 50 ml of Distillate A in a 50-ml Nessler tube. In another similar Nessler tube place 50-ml of water distilled through the apparatus as a control. Add to each tube 0.1 ml of a filtered solution of sodium alizarin sulfonate and 1 ml of freshly prepared hydroxylamine hydrochloride solution and mix well. Add, dropwise and with stirring, either 1 N or 0.05 N sodium hydroxide, depending upon the expected volume of volatile acid distilling over, to the tube containing the distillate until its colour just matches that of the control, which is faintly pink. Then add to each tube 1.0 ml of 0.1 N hydrochloric acid, and mix well. From a burette, graduated in 0.05 ml, add slowly to the tube containing the distillate enough thorium nitrate solution so that, after mixing, the colour of the liquid just changes to a faint pink. Note the volume of the solution added, then add exactly the same volume to the control, and mix. Now add to the control solution sodium fluoride from a burette to make the colours of the two tubes match after dilution to the same volume. Mix well, and allow all air bubbles to escape before making the final colour comparison. Check the endpoint by adding 1 or 2 drops of sodium fluoride to the control. A distinct change in colour should take place. Note the volume of sodium fluoride added.

Dilute Distillate B to 100 ml, and mix well. Place 50 ml of this solution in a 50-ml Nessler tube, and follow the procedure used for Distillate A. The total volume of sodium fluoride required for the solutions from both Distillate A and Distillate B should not exceed 2.5 ml.

E-2 METHOD II ION-SELECTIVE ELECTRODE METHOD

Sodium fluoride solution (5 μg F per ml). Transfer 2.210 g of sodium fluoride, previously dried at 110° for 2 h and accurately weighed, into a 400-ml plastic beaker, add 20 ml of water, and stir until dissolved. Quantitatively transfer this solution into a 1 000-ml volumetric flask with the aid of water, dilute to volume with water, and mix. Store this stock solution in a plastic bottle. On the day of use, transfer 5.0 ml of the stock solution into a 100-ml volumetric flask, dilute to volume with water, and mix.

E-2.1 Calibration Curve

Transfer into separate 250-ml plastic beakers 1.0, 2.0, 3.0, 5.0, 10.0 and 15.0 ml of the *sodium fluoride solution*, add 50 ml of water, 5 ml of 1 N hydrochloric acid, 10 ml of 1 M sodium citrate, and 10 ml of 0.2 M disodium EDTA to each beaker, and mix. Transfer each solution into a 100-ml volumetric flask, dilute to volume with water, and mix. Transfer a 50-ml portion of each solution into a 125-ml plastic beaker, and measure the potential of each solution with a suitable ion-selective electrode apparatus, using a suitable reference electrode. Plot the calibration curve with μg F per 100-ml solution on the logarithmic scale.

E-2.2 Procedure

Transfer 1.00 g of the sample into a 150-ml glass beaker, add 10 ml of water, and, while stirring continuously, add 20 ml of 1 N hydrochloric acid slowly to dissolve the sample. Boil rapidly for 1 min, then transfer into a 250-ml plastic beaker, and cool rapidly in ice water. Add 15 ml of 1 M sodium citrate and 10 ml of 0.2 M disodium EDTA, and mix. Adjust the pH to 5.5 ± 0.1 with 1 N hydrochloric acid or 1 M sodium hydroxide, if necessary, then transfer into a 100-ml volumetric flask, dilute to volume with water, and mix. Transfer a 50-ml portion of this solution into a 125-ml plastic beaker, and measure the potential of the solution with the apparatus described under calibration curve. Determine the fluoride content, in μg, of the sample from the calibration curve.

ANNEX F

[*Table* 1, *Sl No.* (viii)]

TEST FOR MAGNESIUM

F-1 PROCEDURE

Place 400 mg of the sample, 5 ml of diluted hydrochloric acid, and about 10 ml of water in a small beaker, and dissolve the sample by heating on a hot plate. Evaporate the solution to a volume of about 2 ml and cool. Transfer the residual liquid into a 100-ml volumetric flask, dilute to volume with water, and mix. Dilute 7.5 ml of this solution to 20 ml with water, add 2 ml of sodium hydroxide and 0.05 ml of 1 in 1 000 solution of Titan yellow (Clayton yellow), mix, allow to stand for 10 min, and shake. Any colour does not exceed that produced by 1.0 ml of magnesium standard solution (50 μg Mg ion) in the same volume of a control containing 2.5 ml of the sample solution (10 mg sample) and the quantities of the reagents used in the test.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc: No. FAD 8 (716).

Amendments Issued Since Publication

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AMENDMENT NO. 1 FEBRUARY 2005
TO
IS 6031 : 1997 CALCIUM PROPIONATE, FOOD GRADE — SPECIFICATION

(*First Revision*)

[*Page* 1, *Table* 1, *Sl No.* (ii), *col* 3] — Substitute '4.0' *for* '5.0'.

(*Page* 1, *Table* 1) — Insert the following at the end of the table:

Sl No.	Characteristic	Limit	Methods of Test, Ref to	
			Annex of This Standard	Other Standard
(1)	(2)	(3)	(4)	(5)
ix)	Lead (as Pb), mg/kg, *Max*	5	—	Cl 15 of IS 1699

(FAD 8)

Reprography Unit, BIS, New Delhi, India